CONTRACT NO: DAMD17-90-C-0090

TITLE: MIXED-BED, ION EXCHANGE DEVICE FOR WATER PURIFICATION

PRINCIPAL INVESTIGATOR: Michael A. Taylor, Ph.D.

CONTRACTING ORGANIZATION: Sepratech
2131 Las Palmas Dr., Suite A
Carlsbad, California 92008

REPORT DATE: October 1, 1990

TYPE OF REPORT: Phase I Final Report

PREPARED FOR: U.S. ARMY MEDICAL RESEARCH AND DEVELOPMENT COMMAND
Fort Detrick, Frederick, Maryland 21702-5012

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED
	1 October 1990	Phase I Final (4/2/90-10/2/90)

4. TITLE AND SUBTITLE
Mixed-Bed, Ion Exchange Device for Water Purification

5. FUNDING NUMBERS
Contract No.
DAMD17-90-C-0090

65502A
3P665502M802.BA.166
WUDA346137

6. AUTHOR(S)
Michael A. Taylor, Ph.D.

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
Sepratech
2131 Las Palmas Drive, Suite A
Carlsbad, California 92008

8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES)
U.S. Army Medical Research & Development Command
Fort Detrick
Frederick, Maryland 21702-5012

10. SPONSORING / MONITORING AGENCY REPORT NUMBER

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION / AVAILABILITY STATEMENT
Approved for public release;
distribution unlimited

12b. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*

14. SUBJECT TERMS
Ion Exchange; Deionization; Water Purification; RA II; SBIR; Phase I; Water supply

15. NUMBER OF PAGES

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
Unclassified	Unclassified	N/A	N/A

APPENDIX A

U.S. DEPARTMENT OF DEFENSE

SMALL BUSINESS INNOVATION RESEARCH (SBIR) PROGRAM

PROPOSAL COVER SHEET

Failure to fill in all appropriate spaces may cause your proposal to be disqualified.

TOPIC NUMBER: DAMD17-90-C-0090

PROPOSAL TITLE: Mixed-Bed, Ion Exchange Device for Water Purification

FIRM NAME: Sepratech

MAIL ADDRESS: 2131 Las Palmas Dr., Ste. A

CITY: Carlsbad, STATE: CA ZIP: 92009

PROPOSED COST: PHASE I OR II PROPOSAL: I PROPOSED DURATION IN MONTHS: 6

BUSINESS CERTIFICATION:

	YES	NO
▶ Are you a small business as described in paragraph 2.2?	☒	☐
▶ Are you a minority or small disadvantaged business as defined in paragraph 2.3?	☐	☒
▶ Are you a woman-owned small business as described in paragraph 2.4?	☐	☒
▶ Will you permit the government to disclose the information on Appendix B, if your proposal does not result in an award, to any party that may be interested in contacting you for further information or possible investment?	☒	☐
▶ Has this proposal been submitted to other US government agency/agencies; or DoD components, or other SBIR Activity? If yes, list the name(s) of the agency, DoD component or other SBIR office in the spaces to the left below. If it has been submitted to another SBIR activity list the Topic Numbers in the spaces to the right below:	☐	☒

▶ Number of employees including all affiliates (average for preceding 12 months)

PROJECT MANAGER/PRINCIPAL INVESTIGATOR	CORPORATE OFFICIAL (BUSINESS)
NAME: Michael A. Taylor, Ph.D.	NAME: Mark Sizelove
TITLE: Product Development	TITLE: President
TELEPHONE: (619) 438-5233	TELEPHONE: (619) 438-5233

For any purpose other than to evaluate the proposal, this data except Appendix A and B shall not be disclosed outside the Government and shall not be duplicated, used or disclosed in whole or in part, provided that if a contract is awarded to this proposer as a result of or in connection with the submission of this data, the Government shall have the right to duplicate, use or disclose the data to the extent provided in the funding agreement. This restriction does not limit the Government's right to use information contained in the data if it is obtained from another source without restriction. The data subject to this restriction is contained on the pages of the proposal listed on the line below.

PROPRIETARY INFORMATION:

DISCLOSURE PERMISSION STATEMENTS: All data on Appendix A are releasable. All data on Appendix B, of an awarded contract, are also releasable.

Michael A Taylor — SIGNATURE OF PRINCIPAL INVESTIGATOR — 9/1/90 DATE

Mark Sizelove — SIGNATURE OF CORPORATE BUSINESS OFFICIAL — 10-1-90 DATE

Nothing on this page is classified or proprietary information/data.

U.S. DEPARTMENT OF DEFENSE
SMALL BUSINESS INNOVATION RESEARCH (SBIR) PROGRAM
PROJECT SUMMARY

TOPIC NUMBER: DAMD17-90-C-0090

PROPOSAL TITLE: Mixed-Bed, Ion Exchange Device for Water Purification

FIRM NAME: Sepratech

PHASE I or II PROPOSAL: I

Technical Abstract (Limit your abstract to 200 words with no classified or proprietary information/data.)

I. PROJECT SUMMARY

The purpose of this contract was to determine the feasibility of development and subsequent production of a small ion exchange device for water purification in a field setting. The device specifications included: a capacity of 1 g of NaCl; flow rates of 20-25 or 200-300 ml/min; removal of dissolved solids to less than 1 mg/ml; and operation in any orientation without channel formation.

The work performed included selection of a mixed-bed, ion exchange resin combination after determination of working capacities, physical characteristics, and resistance to temperature stresses.

Prototype housing design included analysis of: 1.) device patents, 2.) flow patterns, 3.) bed volume changes, 4.) connection requirements, and 5.) housing materials; Construction included development of: 1.) volume compensating frits, 2.) inlet and outlet covers, 3.) component design, 4.) weld site engineering, and 5.) manufacturing procedures.

Two device designs were used to construct fully functional prototypes with working capacities of 6.5 and 1.0 grams of NaCl. These devices were effective in any orientation without evidence of channel formation at flow rates of 275 and 25 ml/min respectively.

Anticipated Benefits/Potential Commercial Applications of the Research or Development

Validation of the prototype indicated that these devices exceeded the specifications necessary for production of ultra-pure water in any setting. This device could be used as a means of sampling or isolation for any application where elimination of degradative forces could increase sampling accuracy.

List a maximum of 8 Key Words that describe the Project.

Ion Exchange

Deionization

Water Purification

II. DETAILED PROJECT OBJECTIVES

A. PRIMARY OBJECTIVE

The primary objective of this project was to determine the feasibility of designing and constructing a Mixed-Bed, Ion Exchange device with a capacity of 1 gram of Sodium Chloride from source water containing 10 mg/ml of dissolved salts at flow rates up to 300 ml/min. The device must be equally effective in any orientation without the formation of channels within the separatory bed, and must maintain a uniformity of flow to maximize capacity. Included in the primary objective was the construction of working prototypes to exemplify the functional potential and possibilities of mass production of these devices.

B. OBJECTIVES ACCOMPLISHED TO ACHIEVE THE PRIMARY GOAL

1. Ion Exchange resin selection by:
   a. Analysis of resins by:
      1.) Collection of manufacturers published and unpublished data on resin characteristics,
      2.) Analysis of Resin data,
      3.) Determination of the cost effectiveness of resins, and
      4.) Selection of a group of resins for further testing.
   b. Testing of resins by:
      1.) Obtaining test samples of resins, and
      2.) Testing of resins for:
         a.) Working capacity;
         b.) Resistance to temperature stresses at:
            (1.) High temperatures, and
            (2.) Freezing and thawing; and
         c.) Physical changes in separatory bed volume following exposure to dissolved salts.

2. Design the device by:
   a. Determination of whether patented Sepratech, Ion Exchange device designs and technologies could be effectively incorporated into a device designed for field use in the purification of RO pretreated water, and
   b. Designing the device housing by:
      1.) Determination of an appropriate flow design within the housing through:
         a.) Analysis of existing patent designs for determination of the applicability of use in the proposed device,
         b.) Determination of the flow designs necessary to achieve the specified flow rates,
         c.) Determination of the separatory bed volume necessary to achieve a working capacity sufficient to meet the water volume production specifications, and
         d.) Selection of appropriate connections for use in field settings and to allow adequate flow rates to achieve the specified volumes of water output; and

2.) Selection of an appropriate housing material by:
    a.) Analysis of the physical and chemical characteristics of various potential housing materials based upon:
        (1.) Impact strength,
        (2.) Resistance high and low temperature stress,
        (3.) Relative cost,
        (3.) Ease of manufacturing,
        (4.) FDA approval for use in contact with food and medical devices based upon levels of extractibility, and
        (5.) Resistance to physical and chemical stresses associated with use in a field setting, i.e. ozone and ultraviolet irradiation.
3.) Determination of an effective weld site design adequate to withstand the postulated pressures generated within the device during use and exposure to temperatures reached during autoclaving; and

c. Design the housing components, including:
    1.) Selection and development of an effective frit material which compensated for changes in separatory bed volume during operation, and
    2.) Selection and acquisition of effective inlet and outlet covers capable of temperature resistance without welding to the housing ports or loosening due to differences in expansion coefficients.

3. Construct prototype devices by:
    a. Selection of materials for prototype construction,
    b. Preparation of engineering drawings of the prototype,
    c. Manufacture of prototype weld sites, and
    d. Manufacture of prototype housings.

4. Test manufacture of prototype devices to determine effective methods of assembly and construction.
    a. Determination of the most effective welding machine parameters to weld together the housing components, and
    b. Determination of the most effective procedures to fill housing uniformly without gaps, channels, or pockets.

5. Performance of validation testing of the prototype working capabilities; including:
    a. Working flow rate potential during use as the separatory bed changes in volume,
    b. Working capacity, and
    c. Demonstration of the ability of the device to remain effective in any orientation by determination of:
        1.) Working capacity of prone devices
        2.) Working flow rates in the prone position
        3.) Absence of channel formation in devices in the prone position

III. WORK CARRIED OUT

A. ION EXCHANGE RESIN SELECTION

Analysis of available information on Ion Exchange resins

The initial phase of this project was the selection of the Ion Exchange Resins for preparation of the separatory bed. To select the most effective resin, Ion Exchange resin manufacturers were contacted to request published, technical data on any resins with potential for use in water purification. Although all strengths of resins were investigated, it was felt that a Mixed-Bed combination of hydrogen forms of strong acid and free base or hydroxide forms of strong base exchangers provided the highest degree of dissociation, therefore, were most appropriate for this application. The greater degrees on resin dissociation associated with strong ion exchange resins enabled increased, irreversible ionic interactions between resins and contaminating ions within the feedwater. Another reason for the preference of a Mixed-Bed combination of strong acid, cationic and strong base, anionic exchangers was the predetermination that disposability was preferable over regeneration.

The manufacturers contacted included: Syborn Incorporated; Dow Chemical Corp.; Bio-Rad Laboratories, Inc.; Rohm & Haas Co.; Mallinkrodt, Inc.; Pierce Chemical Co.; Sigma Chemical Co.; Aldrich Chemical Co.; J.T.Baker Chemical Co.; Applied Separations Inc.; Benson Polymerics Inc.; ES Industries Inc.; EM Science; Alltech Associates, Inc.; Pharmacia LKB Biotechnology, Inc.; Macherey-Nagel; and Baxter Scientific Products.

The data obtained from ion exchange resin manufacturers were evaluated for the following criteria:

1. Compliance of resin composition with Code of Federal Regulations 21:173.25 for use in preparation of food and medical materials,
2. The highest thermal resistance (for autoclaving and freeze-thawing),
3. The highest capacity,
4. The greatest flow potential,
5. The particle size,
6. The greatest resistance to fracture,
7. The least response to osmotic shock, i.e. swelling when saturated with dissociated ions, and
8. The relative cost per volume.

Selection of resins for further testing

Following completion of the analysis of all available information on investigated resins, the ion exchange resins listed in Table 1. were selected for further testing.

Obtain resin samples for further analysis

Seventeen test resin samples were obtained after persistent requests from 5 of the listed manufacturers. Two samples of particular interest were not received until the project term was near completion. Ecotec, Inc. provided separate cationic and anionic exchange resins. These resins were of interest because they were the smallest sized particle of all industrial resins. In spite of the reduction in particle size the capacity was purported to be unchanged. Sybron provided the mixed-bed resin, Ionac NM-201/SG. This resin combination had very recently been brought to market. This was described as Sybron's highest capacity, nuclear grade of mixed-bed resin.

Table 1. Ion Exchange Resins Selected for Further Testing

Composition	Manufacturer	Resin
Mixed Bed Resins	Dow Chemical	Dowex MR-3
	Rohm & Haas	Amberlite MB-1
	Rohm & Haas	Amberlite IRN-150
	Syborn	Ionac NM-60/60
	Syborn	Ionac NM-201/SG
Strong Acid Cationic Exchanger	Bio-Rad Lab	AG 50X8
	Dow Chemical	Dowex HCR-S
	Rohm & Haas	Amberlite IR-120
	Rohm & Haas	Amberlite IRN-77
	Syborn	Ionac C-267/SG
	Ecotec	Cationic Resin
Strong Base Anionic Exchanger	Bio-Rad Lab	AG 1X8
	Dow Chemical	Dowex HCRW 2
	Rohm & Haas	Amberlite IRN-78
	Rohm & Haas	Amberlite IRA-400
	Syborn	Ionac ASB-1P
	Ecotec	Anionic Resin

Resin Testing

Analysis of the data provided by resin manufactures indicated that, as postulated in the proposal application, additional testing was necessary to select a resin combination capable of meeting the requirements associated with use in field settings. The parameters described were tested for the defined reasons.

The following tests were performed on the resin samples:

Determination of the working capacity of selected resins

The initial capacity testing was designed to be performed under working conditions with fluid passing through a closed vessel rather than in a static condition, as in a slurry.

The standard bead size for resins used in industrial applications, most commonly range from 16 to 50 mesh size or roughly 0.3 to 1.18 mm. These were relatively large particles. Because of their size, the amount of interstitial spaces between the particles was also relatively large. As the system size (particularly bed height) decreased the likehood of the fluid phase passing through the systems without encountering attractive forces associated with the resin proportionately increased. Thus, the capacity of separatory bed was dependent upon the dimension of the housing as well as the flow rate through the system. These characteristics were even more significant in smaller devices with minimal bed height. Therefore, the working capacity under defined conditions were unique to each design and must be determined empirically.

One way of maximizing the capacity of the separatory bed was through the use of smaller resin particles. This was our initial reason for investigating resins commonly used in biotechnology related applications where must smaller

fluid volume were involved.

Determination of resistance to temperature stresses

Resistance to high temperatures

In order to assure optimal use of these devices, they needed the longest shelf-life possible, therefore, must be free of any potential pyrogens. To assure non-contamination during storage these devices were required to be sterilized during manufacture. Therefore, the components and resins must be able to withstand the temperatures and pressures associated with autoclaving or an alternative means of decontamination. Potential problems resulting from exposure to elevated temperatures included: changes in the strength of the housing and the housing weld, loss of Ion Exchange bed capacity, and degradation of the Ion Exchanger support. This testing was particularly important for hydroxyl forms of anion exchangers, because of their sensitivity to diminished capacity at elevated temperatures.

Resistance to freezing

In field settings, potential exposure to temperatures below freezing would be inevitable, therefore, housing and resin testing included exposure to the stresses associated with freezing and thawing. The potential problems associated with frigid temperatures included increased brittleness of the housings and the fracture of the particles of the separatory bed. There was also the potential that the working capacity of the separatory bed would be diminished following freezing.

Determination of the physical changes reins during use

As the dissociated ions from the fluid phase were removed by the Ion Exchangers, the support matrix compresses. This results in diminished bed volume. In order to provide adequate compensation for the bed volume changes to prevent channel formation, the amount of bed volume change must be determined under working conditions. Since the bed volume changes were a function of the working capacity, these characteristics were also dependent upon the device dimensions, the flow rate, and the test conditions. These parameters must be determined empirically.

B. DESIGN THE DEVICE HOUSING

Analysis of the applicability of Sepratech Ion Exchange patents

Because of the similarity in the specifications for the Ion Exchange device solicited in this project and the claims of Sepratech patented design of Ion Exchange devices, it was felt that the patented designs were potentially applicable to the project. However, it was necessary to analyze the designs to determine what modifications were necessary to reach the proposed requirements. Specifically, the flow rates were to be increased, therefore, it was necessary to determine whether the closed system could sustain the defined flow rate. It was also necessary to define whether the backpressure generated within the flow distribution chamber was adequate to maintain a uniformity of flow in larger devices.

Determination of an appropriate flow design

Following review of the patented designs, the design of flow through the

proposed prototype devices was defined. This was determined by review of the limits of flow rate, resulting back pressure, bed diameter, and bed height in relation to the maintenance of the uniformity of flow within the system.

Determination of the housing design adequate for prototype construction

It was necessary to adapt the housing design to provide for ease of construction and assembly of functional prototypes. The ultimate device housing was expected to be constructed of two injection molded pieces, ultrasonically welded together. However, the prototype was constructed of five separate pieces and assembled with four ultrasonic welds.

Determination of the separatory bed volume

Following analysis of separatory bed capacity per bed volume data provided by manufacturers and determined by preliminary testing, the housing internal volume and ratio of height to diameter were defined. These dimensions took into considerations expected differences in working capacities unique to each device and the test conditions.

Selection of appropriate connections for use in field settings

To assure that this device was attached with the other portions of the IV water maker securely, aseptically, and easily, an appropriate means of connecting portion of the system was developed.

Selection of an appropriate housing material

Devices dedicated to medical applications in field settings places special requirements on the materials used to construct these devices. Therefore, an analysis of the physical and chemical characteristics of various potential housing materials was performed. This analysis was based upon:

1. Impact strength
2. Resistance high and low temperature stress
3. Resistance to anticipated working pressures
4. Relative cost
5. Ease of manufacturing
6. FDA approval for use in contact with food and medical devices based upon levels of extractibility.
7. Resistance to physical and chemical stresses associated with use in a field setting, i.e. ozone and ultraviolet irradiation.

Determination of an effective weld site design

Construction of prototypes and the final product required that a weld site be designed, constructed, and validated. This weld site must be capable of withstanding at least 4 times the working pressures postulated to occur within the device. The design had to take into consideration the materials characteristics an housing dimensions to assure weld uniformity and strength. To construct prototypes of this weld site it was necessary to have the cylinders and tops machined to close tolerances (+/- 0.001 inches). Depending upon the material used this could be very difficult, particularly if extruded cylinders were used in prototypes construction. Extruded materials vary considerably in dimensions.

C. DESIGN THE HOUSING COMPONENTS

Selection and development of an effective frit material

Since the separatory bed volume was expected to diminish considerably during operation, the volume compensating frit material must be designed to meet or exceed this decreased volume. This portion of the device design was critical to prevent the formation of channels within the separatory bed. In addition the frit must have sufficient backpressure to force fluids entering the device to the periphery, prior to passing through the frit. This enhances the uniformity of flow within the device. However, the backpressure was not to be excessive in order to minimize the total backpressure within the system. The frits must be composed of materials with FDA/USP approved levels of extractibility and must have resistance to 121 degrees centigrade.

Selection and acquisition of effective inlet and outlet covers

Device use in field settings required that the inlet and outlets be covered securely to prevent contamination. The covers must also be easily removable. This required that they were composed of dissimilar materials. Like materials have a tendency to bind and even weld when exposed to elevated temperatures. The covers had to have compatible coefficients of expansion since temperature variation could result in the loosening of covers.

D. CONSTRUCT PROTOTYPE DEVICES

After the housing design was decided drawings were prepared for use by the contracted machining facilities. Three facilities were contracted to mill prototype weld sites. This enabled comparative analysis of each facilities ability to perform the milling of the complete housings to the desired tolerances. Following selection of a single facility, 75 prototypes were machined from extruded polycarbonate cylinder and flat stock.

E. TEST MANUFACTURE PROTOTYPE DEVICES

In order complete assembly of functional prototypes it was necessary to develop effective methods of assembly and construction, including the following:

Define the most effective welding procedures

For selection of the machine facility for prototype milling and the optimization of ultrasonic welds on prototypes the welding procedures had to defined. The ultrasonic weld of the housings components required the coordination of nine separate parameters. The gain of booster had to be matched with the frequency of the welding horn. In addition the speed of arm travel had to be matched with the weld time, the pretriggering of the weld cycle, the hydraulic pressure of the welding arm, the welder triggering pressure, and the end of weld shut-off. It was necessary to determine the majority of these settings empirically through progressive adjustments, weld inspection, and pressure testing.

Determination of the effective cylinder filling procedures

For effective function the housing had to be filled uniformly without gaps, channels, or pockets. Several basic methods and numerous variations

were tested, including use of dry or wet (slurried) particles.

F. VALIDATE THE COMPLETED PROTOTYPE FUNCTIONAL POTENTIAL

Determination of the working flow rate

The working flow rate and associated backpressures in various orientations were determined for both sized prototypes with several different separatory beds. This testing was monitored as the separatory bed volume changed to monitor for potential breakthrough due to channel formation.

Determination of the working capacity

The working capacity is various orientations was determined for both sized prototypes with several different separatory beds. This testing was monitored as the separatory bed volume changed to monitor for potential breakthrough due to channel formation.

IV. RESULTS OBTAINED

A. RESIN COST ANALYSIS

Cost Analysis of Resins

The cost of the resins commonly used for industrial applications ranged from $82 to $84 per cubic foot for anionic exchangers and $218 to $226 per cubic foot for cationic exchangers. Performed mixed exchangers were from $151 to $165. Resins of the same support composition i.e. styrene linked divinylbenzene, used in laboratory-scale systems were priced at $45 to $52 per 100 grams. This resin, swelled at ionic saturation had a bed volume of 150 ml per 100 grams. Therefore, the price per cubic foot calculated to be roughly $9800 per cubic foot. The later resins are also of considerably more fragile structure, therefore, these resins were not felt to be appropriate for this application.

We were unable to locate industrial resin suppliers manufacturing particles smaller than the 16 to 50 mesh size, with the single exception of Ecotec Inc. Their resin sizes were from 100 to 200 mesh. Because their processes reduce resin size without changing the per particle capacity, the relative capacity of an equivalent bed volume was postulated to be markedly increased.

B. RESIN TESTING

Capacity testing

The baseline working capacity of resin samples was initially tested in closed, 5 ml vessels at slow flow rates between 20 to 30 ml/min. Weighed sample of resin were exposed to source water consisting of ultra-pure water containing 100 mg/l of Sodium Chloride. The effluent from the test device was monitored for changes in TDS. The solution was recirculated through the test sample. Increased TDS was taken to indicate saturation of the Ion Exchange resins in this test system under the defined conditions. From the volume of water passing through the device the capacity of the resin was calculated.

The Rohm & Haas, Amberlite IRN-150 and the Sybron, Ionac NM-201/SG had equivalent capacities 23 mg of NaCl per ml of resin at test device saturation (Table 2.). All other resin samples had lower capacities. The ability to

remove dissociated ions increased as the flow rates were decreased.

Table 2. WORKING CAPACITY TESTING

RESIN	BED TYPE	GRADE	WORKING CAPACITY g ions/g resin	g ions/ml resin
AMBERLITE MB-1	MIXED		0.030	0.020
AMBERLITE IRN-150	MIXED	NUCLEAR	0.033	0.022
DOWEX MR-3	MIXED	NUCLEAR	0.030	0.020
DOWEX MRS-C	MIXED		0.025	0.017
IONAC NM-60/SG	MIXED	NUCLEAR	0.025	0.017
IONAC NM-201/SG	MIXED	NUCLEAR	0.032	0.021
ECOTEC (REGENERATED)	MIXED		0.005	0.003
AMBERLITE IR-120	CATIONIC-	NUCLEAR	0.075	0.050
AMBERLITE IRN-77	CATIONIC-H		0.095	0.064
DOWEX HCR-S	CATIONIC-H		0.055	0.037
IONAC C-201/SG	CATIONIC-H		0.086	0.058
ECOTEC	CATIONIC-H		ND	ND
AMBERLITE IRN-78	ANIONIC-O	NUCLEAR	0.065	0.044
AMBERLITE IRA-400	ANIONIC-OH		0.075	0.050
DOWEX SBR-P	ANIONIC-OH		0.053	0.036
IONAC ASB-1P	ANIONIC-OH		0.060	0.040
ECOTEC	ANIONIC-OH		ND	ND

Defining a capacity per unit volume provided what was likely an inherently erroneous basis for comparison. It was clear that the resin capacity was a relative measure of the mix of particle sizes within a given range, the regeneration level, and the level of hydration. The particle size in the 16 to 50 mesh range are from 1.18 millimeters to 0.3 millimeters. Any skewing in the distribution made a significant difference in the bed volume to capacity ratio. The regeneration level depended upon the manufacturer, however, was also dependent upon the age of the resin with labile chemistries (hydroxyl anion exchangers). The level of hydration affected the particle size, therefore, the bed volume. This of course varied considerably as the dissociated salts were immobilized. For these reasons the capacity per bed volume was likely to vary significantly depending upon the conditions.

The working capacity of the Ecotec resin combination was purported to be roughly 6 times the working capacity of the Amberlite IRN-150 and Ionac NM-201/SG samples. The working capacity was based upon assumptions provided by the manufacturer, related to equivalents of absorption of metal ions in solution.

The variation in manufacturers data and the observed capacities were in part attributed to the size of the test vessel and test conditions. Under all manufacturers suggested test systems the bed height was defined as a minimum of 30 inches tall, with flow rates greater than a gallon/minute (3.8 liters/min).

High Temperature Testing

Following exposure of weighed resin samples to 121 degrees Centigrade for 15 minutes in pressurized steam, no observable physical changes were evident (Table 3.), however, there was some loss of working capacity. The results of testing of the separate cation and anion exchangers indicated that no significant loss of capacity occurred among any of the the cationic exchangers. The capacity loss among anionic and mixed-bed exchangers ranged from 5 to 15 percent. In addition, since separate samples of all the constituents of the mixed bed combinations were not available for all samples, it was not possible to verify that the loss of capacity was due entirely to the loss of capacity of the anion exchangers.

Freeze-Thaw Testing

Weighed samples of resins were exposed to -20 degrees Centigrade for 12 hours then allowed to return to room temperature. After thawing fractured resin particles were evident in most of the resin samples (Table 3.). These fractured particles were of a wide range in sizes. Analysis of the particles indicated that the fractured particle sizes were always greater than 100 microns. The fractured particles constituted a very small fraction of the tested volume. Among the 16 to 50 mesh samples weighed samples of particles less than 300 microns was never greater than 0.5% of the bed weight. The amount of fractured particles observed among the Ecotec resins was less than with other resins. No particles less than 75 microns were observable in these test samples. Since the porosity of the media restraints is roughly 20 microns no fractured articles from any samples penetrated the restraints. This parameter was, therefore, not included as a basis for resin selection.

No changes in working capacity were observed among test samples exposed to below zero temperatures (Table 3.)

Resin Swell Testing

Because each sample of resin had varied degrees of hydration with various hydration fluids, the swelling characteristics of each resin were compared between identical, 5 gram samples of resins. One resin sample was hydrated in excess ultra-pure water containing less than 1.0 mg/l of total dissolved water (TDS). The other weighed sample was hydrated in excess 1 M Sodium Chloride. Both samples were allowed to equilibrate for several hours with occasional mixing. Thereafter, the bed volume of each sample was determined. The mean results were listed in Table 4.

The greatest degree of variation in bed volume was seen among the anion and mixed bed exchangers, which was roughly double that of the cation exchangers. This was somewhat surprising since it was initially postulated that the greatest variation would occur within the anion exchangers. It was further postulated that the change in bed volume of the mixed bed exchangers would be roughly the average of the bed volume changes of the single resin samples. This was proposed since the mixed bed exchangers were composed of combinations of the individual cation and anion resins tested. It was of note that the only mixed bed sample not to follow this pattern was the Eco-Tec mixed bed resin. This sample was produced by combining equivalent weighed portions of the individual Eco-Tec cation and anion resins. Also of note was the fact that the weighed samples of the Eco-Tec cation resins had a smaller bed volume than the other manufacturers resins. This could have been accounted for by the considerably smaller particle size of the resin. This

Table 3. Effects of Temperature Stress on Resin Samples

RESIN	BED TYPE	GRADE	CHANGES PHYSICAL TEMPERATURE HIGH	CHANGES PHYSICAL TEMPERATURE LOW	PARTICLE SIZE (um)	% BY WEIGHT	CAPACITY TEMPERATURE HIGH (% OF ORIG.)	CAPACITY TEMPERATURE LOW (% OF ORIG.)
AMBERLITE MB-1	MIXED		NC	FINES	> 100	< 0.5	87	NC
AMBERLITE IRN-150	MIXED	NUCLEAR	NC	FINES	> 100	< 0.5	95	NC
DOWEX MR-3	MIXED	NUCLEAR	NC	FINES	> 100	< 0.5	92	NC
DOWEX MRS-C	MIXED		NC	FINES	> 100	< 0.5	85	NC
IONAC NM-60/SG	MIXED	NUCLEAR	NC	FINES	> 100	< 0.5	86	NC
IONAC NM-201/SG	MIXED	NUCLEAR	NC	FINES	> 100	< 0.5	92	NC
ECOTEC (REGENERATED)	MIXED		NC	NC	> 75	< 0.5		NC
AMBERLITE IR-120	CATIONIC-	NUCLEAR	NC	FINES	> 100	< 0.5	NC	NC
AMBERLITE IRN-77	CATIONIC-H		NC	FINES	> 100	< 0.5	NC	NC
DOWEX HCR-S	CATIONIC-H		NC	FINES	> 100	< 0.5	NC	NC
IONAC C-267/SG	CATIONIC-H		NC	FINES	> 100	< 0.5	NC	NC
ECOTEC	CATIONIC-H		NC	FINES	> 100	< 0.5	NC	NC
AMBERLITE IRN-78	ANIONIC-O	NUCLEAR	NC	FINES	> 100	< 0.5	95	NC
AMBERLITE IRA-400	ANIONIC-OH		NC	FINES	> 100	< 0.5	ND	ND
DOWEX SBR-P	ANIONIC-OH		NC	FINES	> 100	< 0.5	85	NC
IONAC ASB-1P	ANIONIC-OH		NC	FINES	> 100	< 0.5	86	NC
ECOTEC	ANIONIC-OH		NC	FINES	> 100	< 0.5	ND	NC

ND = NOT DONE
NC = NO CHANGE; LESS THAN 5% DIFFERENCE

Table 4. Swell Characteristics of Resins Samples

Resin	Type	Ultra-pure Water	1 M NaCl	% Change
AMBERLITE IRN-77	CATION	6.6	5.75	-12.9
AMBERLITE IR-120	CATION	6.3	5.85	-7.5
IONAC C-267/SG	CATION	6.5	5.9	-9.2
IONAC CFP-110	CATION	6.6	6.0	-9.0
DOWEX HCR-S	CATION	6.5	6.0	-7.7
ECO-TEC	CATION	5.8	5.2	-10.3
AMBERLITE IRN-78	ANION	7.3	5.3	-27.4
IONAC ASB-1P/SG	ANION	7.4	5.5	-25.7
IONAC A-641	ANION	7.0	5.4	-22.9
DOWEX SBR-OH	ANION	7.5	5.8	-22.0
ECO-TEC	ANION	7.8	6.6	-15.4
AMBERLITE IRN-150	MIXED	7.3	5.95	-18.5
AMBERLITE MB-1	MIXED	8.4	5.85	-30.4
IONAC NM-60/SG	MIXED	7.9	5.85	-25.9
DOWEX MRS-C	MIXED	8.4	5.85	-22.0
ECO-TEC	MIXED	6.6	5.9	-10.6

would suggest that Eco-Tec cation exchanger would have relatively greater exposed surface area than other cation exchangers. In contrast, the weighed sample of the Eco-Tec anion exchanger had a greater bed volume than the other resins, in spite of the smaller particle size. This would suggest that this resin had a greater degree of porosity than all other anion exchangers. If the structural integrity were maintained this may facilitate flow rate.

Final Resin Selection

Based upon the results of analysis of manufacturers data, resin testing and cost analysis, three resin combinations were selected to be incorporated into prototype devices. These mixed-bed resin combination are Rohm & Haas, Amberlite IRN-150; the Sepratech prepared Mixed-Bed combination of Ecotec anion and cation exchangers; and Sybron, Ionac NM-201/SG. These resins were selected because of the relatively low backpressure at working flow rates, the working capacity in smaller test devices, the capacity following exposure to elevated temperatures, the resistance to fracturing at low temperatures, the absence of impurities associated with the nuclear grade, and the cost. Although the Ecotec resins did not demonstrate the working capacity anticipated, it was felt that the particle size and the probability of enhancement of the working capacity justified further investigation.

C. HOUSING DESIGN

Flow Design

Existing patented designs of similar devices were reviewed with regards to the design requirements (internal pressure tolerance and potential flow rate) associated with either of the proposed reverse osmosis purification systems. This review indicated that existing flow designs and housing pressure resistance potentials could easily be applicable for use in prototype devices of the approximate size proposed within this contract.

In the device flow design (refer to Fig.1-4) the fluid enters through the inlet and is dispersed to the periphery within the fluid dispersal chamber. This dispersal is induced by the backpressure inherent in the frit. The fluid passes through the frit and into the separatory bed to the downstream frit. Fluid passing through the frit enters the fluid collection chamber and exits the device via the outlet. By forcing the fluid to the periphery, the fluid passes through the device in uniformily; meaning the face of the fluid in the separatory bed is not retarded at the areas away from the inlet and outlet. The uniformity of flow maximizes the capacity of the separatory bed through maximal exposure of the resin to the fluid. In addition, the uniformity of flow enables the the device to be used in any orientation. Flow through the bed is also maintained as the bed volume contracted by frits designed to expand, compensating for volume changes and preventing channel formation.

Housing Design

The prototype housing was decided to consist of three major components, a cylinder and two end pieces. The resin bed was to be contained within the cylinder. The resin was to be prevented from existing the cylinder by upstream and downstream frits. These frits were also to have the capacity to compensate for changes in bed volume as the resin bound free ions within the fluid as it passed through the device. Upstream and downstream of the resin restraining frits were to be fluid dispersal and collection chambers. These

chambers allowed fluid to pass through the device without the generation of vortices or dead spaces, while maximizing the resin exposure to the fluid. Entry and exit were to occur through an inlet in the top and an outlet from the base of the cylinder.

It was determined that in order for the flow to the distal portions of the dispersal chamber the height of this chamber should have been extended. To determine the internal dimensions and resulting flow rates and back pressures of the prototype cylinders, other factors would have to be defined, i.e. capacity/bed volume and relative swell/bed volume.

Housing Internal Volume

The results of working capacity test indicated that to achieve a production capacity of 100 liters, the required separatory bed volume for a 16-50 mesh bead ion exchanger, had to be roughly 150 ml and 25 ml at flow rates of 200 to 300 ml/min and 25 ml/min, respectively. This included enough excess volume to provide reasonable assurance that the proposed production capacity was met.

The most effective volume compensating frit design constructed, had a expanded volume sufficient to compensate for a 20% reduction in total bed volume. If necessary this could have been increased. The nominal height of this frit was 0.42 inches in the larger prototype device.

The outer dimensions of the housing also had to take into consideration the thickness of the housing materials for each of the end pieces, 0.125 inches each.+

To meet these criteria, the prototype housings were constructed to be 5.5 inches in height by 1.5 inches in diameter and 1.75 inches in height and 1 inch in diameter.

Attachment Sites

To afford ease of attachment, resistance to contamination, and pressure resistance, the upstream and downstream attachment sites of the prototype device were decided to include female Leur-lock fittings centrally located at both ends. For construction of prototypes; injection molded, fittings from analogous devices were ultrasonically welded to machined prototype tops.

Tests of the flow rate through the attachment site orifices and the associated back pressures were less than 0.5 PSI at 345 ml/min therefore were acceptable.

Housing Material Selection

Preliminary investigation of the potential housing materials indicated that either polypropylene or polycarbonate were most appropriate materials for prototype construction. Polypropylene provided ease of machining, however, was considerably more difficult to ultrasonically weld effectively. Polycarbonate was more easily welded and was more transparent, but was likely to be more difficult to machine to close tolerances. Polypropylene or polycarbonate devices with wall thickness of 0.125 inches could withstand system pressures up to 150 PSI. The decision was made to use polycarbonate for construction of all prototypes housing components.

Weld Sites

Four weld sites were necessary (Fig.5). These sites were designed to be identical. The weld sites on the cylinder ends and the two end pieces top

were decided to be shear welded together. The weld sites had at least 0.035 inches of interference initiated at the contact point by a 45 degree angle. The tolerances between adjoining outer and inner diameters were milled to within +/- 0.001 inches to provide overall tolerances of +/- 0.002 inches.

Wall Thickness Necessary for Pressure Resistance

Prototype devices of polycarbonate were constructed of materials 0.125 inches in thickness. This was deigned to be more than adequate to endure the projected internal pressures encountered within the envisaged system. In addition this provided a significant level of impact resistance to the device.

D. COMPONENT DESIGN

Frit Material Selection

Initially, polypropylene was preselected as the exclusive component of the frit. However, further analysis indicated that considerably greater volume compensation was required than possible from existing polypropylene frits. Subsequently, a number of combinations of materials were investigated, including multiple nylon mesh, cellulose based mesh, and polypropylene mesh. In addition the initially tested frits generated a pressure drop 5.2 PSI.

A combination of multiple layers of cellulose, nylon, and polypropylene were determined to provide sufficient volume compensation while minimizing the associated pressure to less than 2.0 PSI. The downstream frit of both device were designed to be 0.145 inches in height. The upstream frit of the 150 ml device was designed to provide a minimum expanded height of 0.775 inches and a compressed height of 0.275 inches.

Port Covers for Leur Attachment Sites

Both polypropylene and polycarbonate covers for male and female Leur-lock ports. In order to prevent binding of the covers to the housing dissimilar plastic covers were used. The composition of these covers were approved by FDA/USP for medical use.

E. PROTOTYPE CONSTRUCTION

Prototype Weld Site Construction

Construction of prototypes progressed in a series of steps. The first step was to design, construct, optimize, and test the weld site between component parts of the housing. Preliminary prototype weld sites of several diameters and thicknesses of cylinders were constructed of polycarbonate. This material was selected because of the ease with which it can be welded. Because of the relative rigidity of this plastic, it provided some difficulty in machining. However, since the envisaged device was proposed to be injection molded, it was felt that providing a solution to the welding problems would easiest to begin with polycarbonate prototypes rather than alternative plastics, i.e. polypropylene. Weld sites were milled into cylinders and tops of various diameters. In these prototypes, connections consisted of injection molded, female Leur-lock fittings were welded to prototype tops. Ultrasonic welding equipment was also constructed to test weld these housing. This included bases to secure the components in the proper orientation and welding horns tuned to the frequencies necessary for each weld.

Test procedures were standardized for the different wall thicknesses and

cylinder diameters as well as nine variables associated with the use of the ultrasonic welder. Following optimization of the equipment and welding procedures the welded housings were pressure tested with compressed gas. The defined weld design and procedures withstood internal pressures greater than 100 PSI.

Design and Construction of Prototypes

The two different sized prototype housings were machined from five parts: a single cylinder, two end pieces, and two female Leur-lock fittings. All materials were of 0.125 inch thick polycarbonate. The weld sites were milled to the specifications described in Figure 1. and were within tolerances of 0.002 inches. The same weld procedures were followed as determined in weld testing.

Selection of Filling Procedures

Preliminary testing indicated that achieving a uniform mixture of mixed-bed components, particularly with slurried particles, may be rather difficult. However, this suggested that filling housings with slurried resins may lead to separation of the anion and cation exchangers. The procedures selected included manual filling and compacting to defined pressures of weighed resin aliquots. The 150 ml prototypes were filled with 100 mg of resin. The smaller prototypes were filled with 25 mg of resin.

D. PROTOTYPE VALIDATION TESTING

Flow rate

Both the large and small prototype devices were capable of funtioning at the specified flow rates, roughly 275 and 25 ml/min respectively. There was considerable variation in the backpressure associated with different separatory beds and frit configurations.

Flow rate testing indicated that even though the Ecotec resin was the smallest particle size of all resins samples tested, the backpressure at the specified flow rates were the least of all resins tested. Because of this contradiction, it was decided that this resin should be further investigated. Capacity testing indicated that more complete regeneration of this resin to the hydroxyl derivative was necessary. Efforts to attain the potential capacity indicated by the manufacturer were unsuccessful.

Working Capacity Testing

The working capacity of prototypes of various separatory beds are indicated in Table 5. No evidence of channel formation was observed during any working capacity testing. The curves of ion removal from source water were consistently repetitive until the ion breakthrough occurred. The flow rates and back pressures were consistent, without detectable variations in flow rate.

Table 5. Working Capacity of Completed Prototypes

Separatory	Bed Volume (ml)	Flow Rate (ml/min)	Source Water (mg/l)	Working Capacity (g/device)	Back Pressure (PSI)
R & H IRN-150	150	275	1000	6.6	5.7
Ionac NM-201/SG	150	275	1000	5.2	6.7
Dowex MR-3-C	150	275	1000	5.1	6.0
Ecotec	150	275	N.D.	N.D.	5.1
IR & H IRN-150	25	25	100	1.0	3.0
R & H IRN-150	150	275	10	6.5	5.7
R & H IRN-150	25	25	10	1.0	2.8

UNIT DESIGN CHARACTERISTICS

Large unit

- Solicited Goals: Adaptable to higher flow rate RO pump/filter combination
  - Flow Rate: 200-300 ml/min
  - Capacity: 1 g of NaCl
  - Source Water: 10 mg/ml TDS
  - Dimensions: Within 23 x 20 x 8 centimeters
- Design Characteristics
  - Exterior dimensions
    - Height: 5.5 inches/ 14.0 centimeters
    - Diameter: 1.75 inches/ 4.45 centimeters
    - Bed volume: 150 ml
  - Separatory Matrix
    - Composition: 100 g Rohm & Haas; IRN-150, Nuclear Grade, Mixed-Bed Exchangers
    - Connections: Inlet and Outlet female Leur-lock connectors
    - Housing Materials: Polycarbonate
    - Matrix Restraints: Combinations of Cellulose, Polypropylene, Polyethylene, and Nylon
- Functional capability
  - Weld strength:
  - Working Capacity: 6.6 grams of NaCl (at 285 ml/min)
  - Efficacy in altered orientation

Small Unit

- Solicited Goals:
  - Flow Rate: 20-25 ml/min
  - Capacity: 1 g of NaCl
  - Source Water: 10 mg/ml TDS
  - Dimensions: Within 23 x 20 x 8 centimeters

Design Characteristics

Exterior dimensions

Height:	2.0 inches/ 5.1 centimeters
Diameter:	1.0 inches/ 2.5 centimeters
Bed volume:	6.6 ml

Separatory Matrix

Composition:	12.5 g Rohm & Haas; IRN-150, Nuclear Grade, Mixed-Bed Exchangers
Connections:	Inlet and Outlet female Leur-lock connectors
Housing Materials:	Polycarbonate
Matrix Restraints:	Combinations of Cellulose, Polypropylene, Polyethylene, and Nylon

Functional Capabilities

Weld strength:	
Working Capacity:	1.0 grams of NaCl (at 125 ml/min)
Efficacy in altered orientation	

The following factors are all interrelated in a very complex manner and have a particularly significant effect on small Ion Exchange systems.

5. ESTIMATES OF TECHNICAL FEASIBILITY

Mass production is quite feasible based upon:

1. Completion of two fully functional prototype designs,
2. Demonstrated capabilities which exceed the specified goals for capacity of Sodium Chloride in solution and functional requirements, and
3. Completion of proposed device housing designs, including:
   a. Injection molded cylinder similar to the prototype
      1.) Radial fins in place of multi-layered mesh to support the frits and improve fluid collection.
      2.) Either male or female Leur-lock connection to ASMI standards
   b. Injection molded top
      1.) Fluid collection chamber with minimal space
      2.) Female Leur-lock connection to ASMI standards

The following areas could be improved further in massed produced devices:

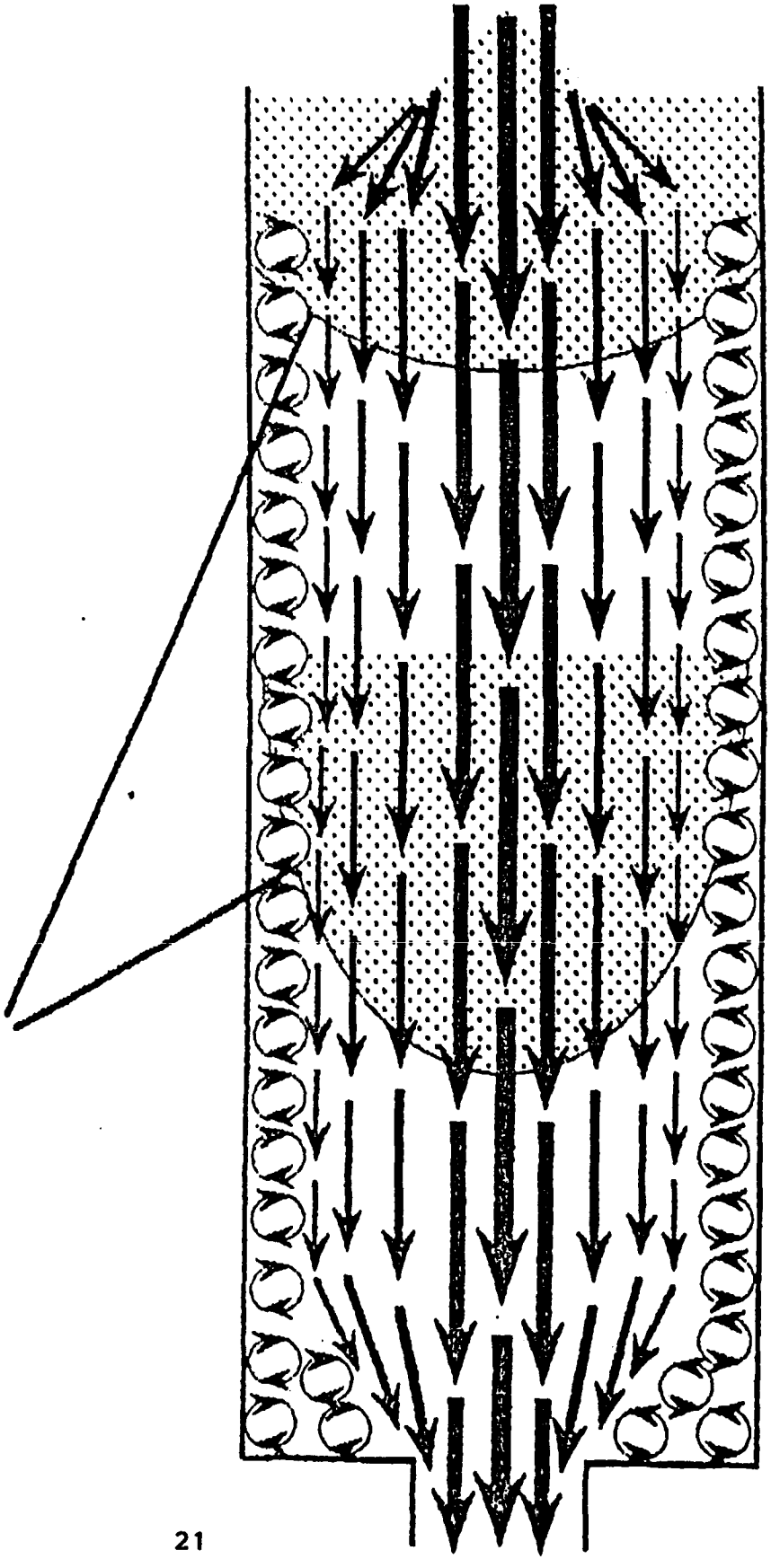
1. Obtaining smaller mesh resin particles. Easily solved with more complete regeneration of existing smaller mesh resins. There is a tendency for some non-uniformity of flow in prototype device with the largest particle sizes.

2. The relative density of counterions immobilized to individual resin particles varies significantly enough to constitute a potential problem, if the water source contains increased concentrations of dissolved salts. The localized attachment of ions can induce localized changes in the separatory bed, i.e. formation of pockets of compacted resins. This was observed only when the source water TD concentration was 1 gram/liter or greater.

3. Obtaining frits of FDA approved materials capable of with standing autoclaving for sterilization.

Figure 1. Flow Through A Tradational Column

Conventional Open Column



Face of Fluid Phase within the device is retarded at the periphery

Sepratech Column

Figure 2 . Sepratech Flow Design

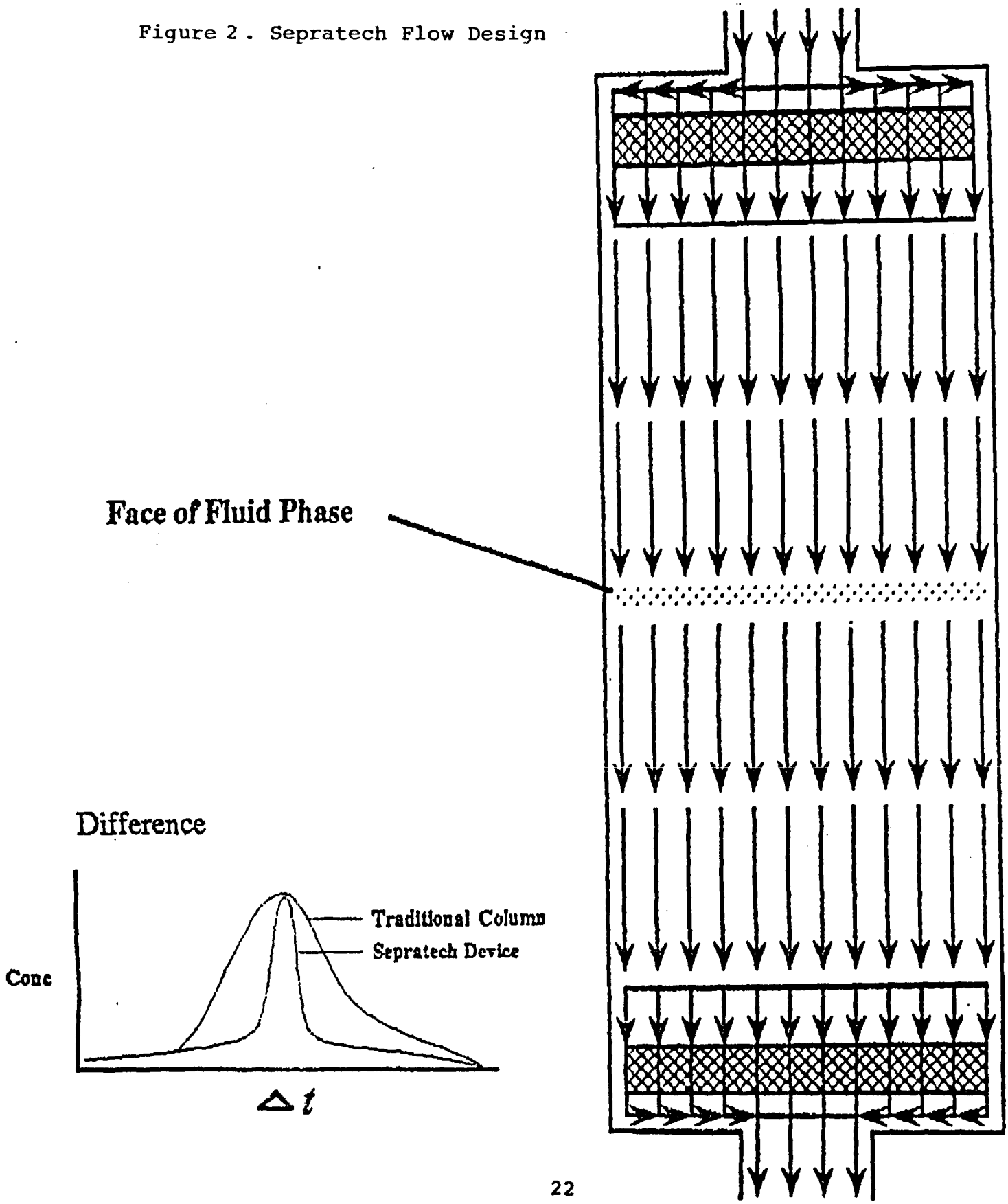


Figure 3 . Bed Volume Compensation

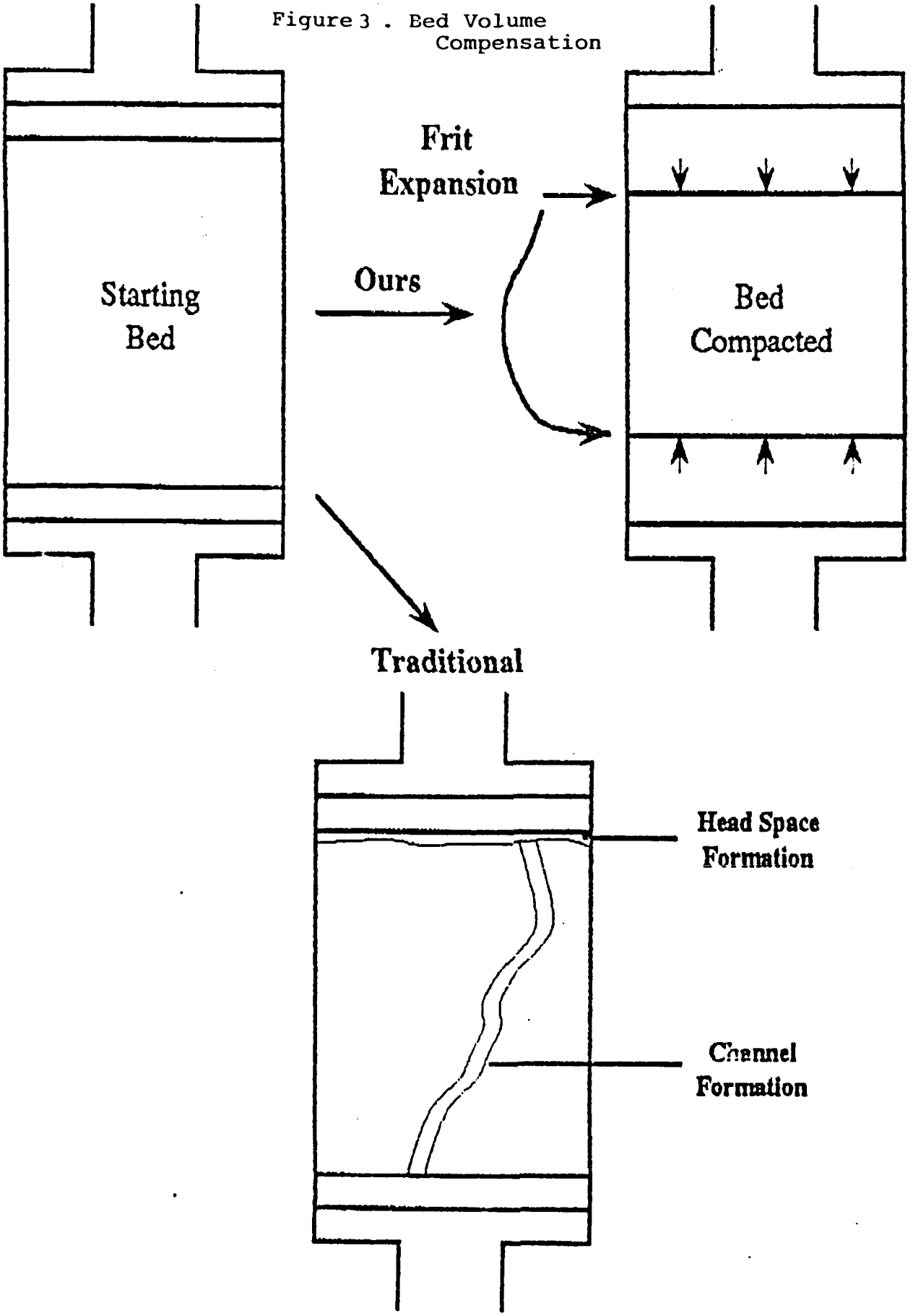
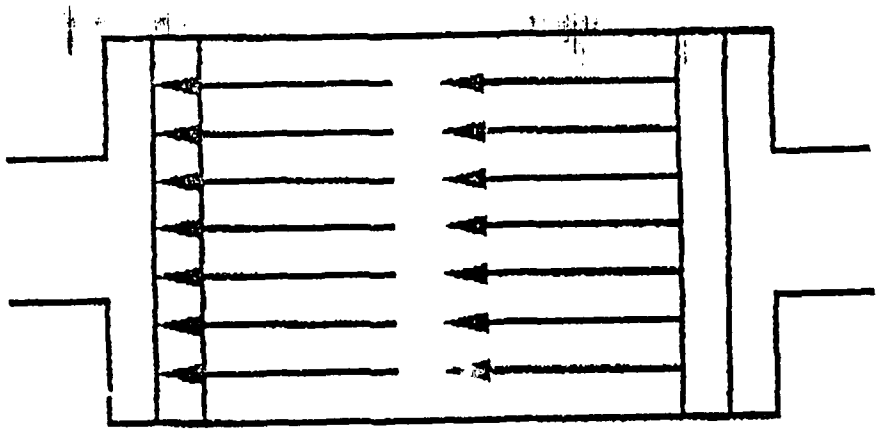


Figure 4. Variability of Orientation

Additional Versatility of Orientation

Ours



Result

- *Decreased Capacity*
- *No Uniformity of Flow*
- *No Separation of Peaks*

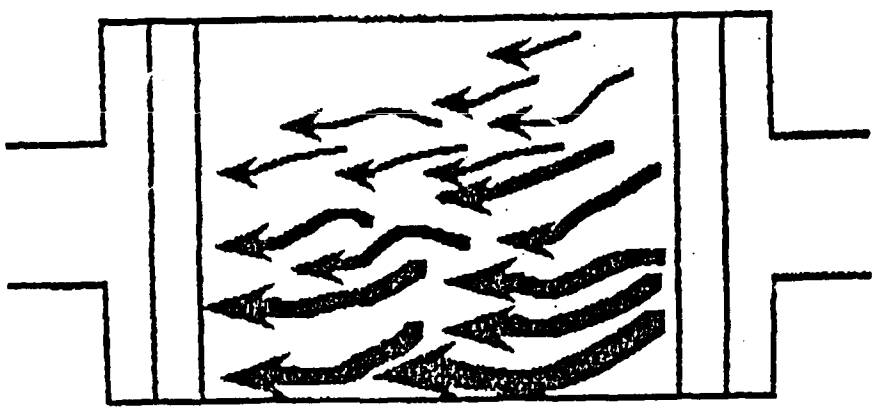


Figure 5. Prototype Housing Desgin